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SILICON PRODUCTION PROCESS EVALUATIONS

QUARTERLY TECHNICAL PROGRESS REPORT (III)

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ABSTRACT

Chemical engineering analysis was continued for the HSC process (Hemlock Semiconductor Corporation) in which solar cell silicon is produced in a 1,000 MT/yr plant.

Progress and status are reported for the primary engineering activities involved in the preliminary process engineering design of the plant: base case conditions (96%), reaction chemistry (96%), process flow diagram (85%), material balance (85%), energy balance (60%), property data (60%), equipment design (40%), major equipment list (30%) and labor requirements (10%).

Engineering design of the second distillation column (D-02, TCS column) in the process was completed. The design is based on a 97% recovery of the light key (TCS, trichlorosilane) in the distillate and a 97% recovery of the heavy key (TET, silicon tetrachloride) in the bottoms. At a reflux ratio of 2, the specified recovery of TCS and TET is achieved with 20 trays (equilibrium stages, N=20). Respective feed tray locations are 9, 12 and 15 ($NF_1 = 9$, $NF_2 = 12$, and $NF_3 = 15$). A total condenser is used for the distillation which is conducted at a pressure of 90 psia.

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I. CHEMICAL ENGINEERING ANALYSIS

Chemical engineering analysis of the HSC process (Hemlock Semiconductor Corporation) was continued. Progress and status for the chemical engineering analysis are summarized below for the primary engineering activities:

		Prior	Current
1.	Base Case Conditions	85%	96%
2.	Reaction Chemistry	85%	96%
3.	Process Flow Diagram	60%	85%
4.	Material Balance	60%	85%
5.	Energy Balance	30%	60%
6.	Property Data	30%	60%
7.	Equipment Design	20%	40%
8.	Major Equipment List	10%	30%
9.	Labor	0%	10%

Status details for the chemical engineering analysis are given in Table I-1. The preliminary process engineering design is based on a 1,000 MT/yr plant for solar cell grade silicon.

Engineering design of the second distillation column (D-02, TCS column) in the process was completed. The function of the distillation column is to separate TCS (trichlorosilane) and TET (silicon tetrachloride). The distillation column has three feeds (F1, F2 and F3):

1. F1 - redistribution reactor effluent
2. F2 - chlorosilanes from the recovery unit
3. F3 - bottoms from the initial distillation (D-01, stripper column)

The TET in the bottoms from the distillation is recycled to the hydrochlorination reactor for additional conversion. The TCS from the distillation is sent to the boron removal unit and a subsequent additional distillation.

Specifications for performing the process engineering design of D-02 distillation column include a 97% recovery of the light key (TCS, trichlorosilane) in the distillate and a 97% recovery of the heavy key (TET, silicon tetrachloride) in the bottoms. A total condenser is used for the distillation which is conducted at a pressure of 90 psia. Additional specifications including detailed feed stream data (flows and concentrations: F1, F2, F3, X_{F1}, X_{F2} and X_{F3}) are given in Appendix A1.

Process engineering results for the design of D-02 are summarized in Appendix A2. At a reflux ratio of 2, the specified separation of 97% recovery of TCS and TET is achieved with 20 trays (equilibrium stages, N= 20). Respective feed tray locations at this reflux ratio are 9, 12 and 15 (N_{F1} = 9, N_{F2} = 12 and N_{F3} = 15).

Additional results including distillate and bottoms data (flow and concentration) for the distillation are given in the summary tabulation.

The design curve for D-02 distillation column is shown in Figure I-1 which presents the results for number of trays (equilibrium stages) required for the distillation. The design curve discloses the variation of required number of trays with reflux ratio for the distillation.

Engineering design of the third distillation column (D-03, DCS column) in the process was initiated during this reporting period. The process engineering calculations now in progress will be presented in the next report.

Table I-1

CHEMICAL ENGINEERING ANALYSIS:

PRELIMINARY PROCESS DESIGN ACTIVITIES FOR HSC PROCESS

Prel. Process Design Activity	Status	Prel. Process Design Activity	Status
1. Specify Base Case Conditions	◐	6. Property Data	◐
1. Plant Size	◐	1. Physical	◐
2. Product Specifics	◐	2. Thermodynamic	◐
3. Additional Conditions	◐	3. Additional	◐
2. Define Reaction Chemistry	◐	7. Equipment Design Calculations	◐
1. Reactants, Products	◐	1. Storage Vessels	◐
2. Equilibrium	◐	2. Unit Operations Equipment	◐
		3. Process Data (P, T. rate, etc.)	◐
3. Process Flow Diagram	◐	4. Additional	◐
1. Flow Sequence, Unit Operations	◐		
2. Process Conditions (T, P, etc.)	◐	8. List of Major Process Equipment	◐
3. Environmental	◐	1. Size	◐
4. Company Interaction (Technology Exchange)	◐	2. Type	◐
		3. Materials of Construction	◐
4. Material Balance Calculations	◐	9. Production Labor Requirements	◐
1. Raw Materials	◐	1. Process Technology	◐
2. Products	◐	2. Production Volume	◐
3. By-Products	◐		
		10. Forward for Economic Analysis	O
5. Energy Balance Calculations	◐		
1. Heating	◐		
2. Cooling	◐		
3. Additional	◐		

O Plan
◐ In Progress
● Complete

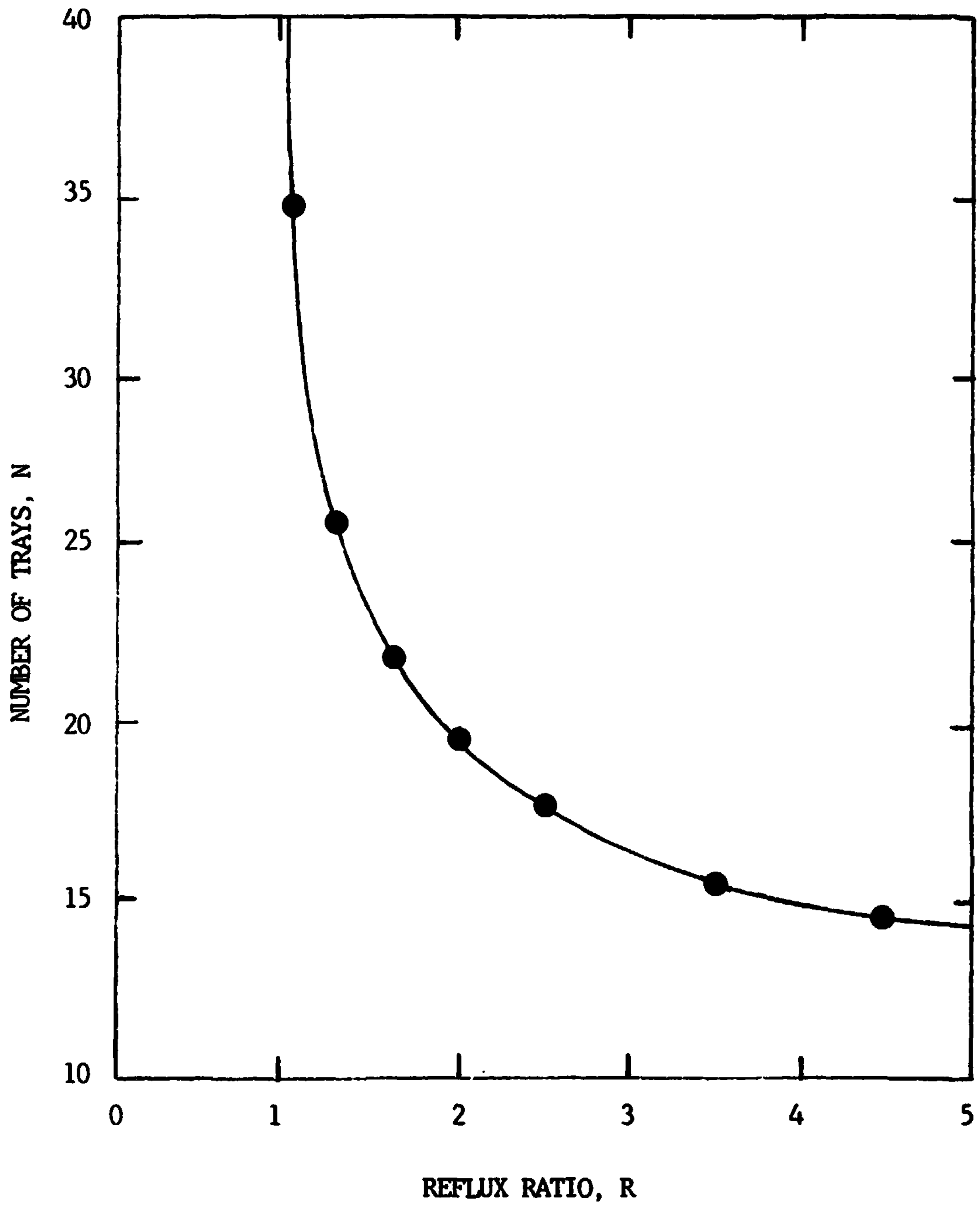


Figure I-1 Design Curve for Distillation, D-02

II. SUMMARY - CONCLUSIONS

The following summary-conclusions are made as a result of achievements during this reporting period:

1. Chemical engineering analysis was continued for the HSC process (Hemlock Semiconductor Corporation) in which solar cell silicon is produced in a 1,000 MT/yr plant.

2. Progress and status are reported for the primary engineering activities involved in the preliminary process engineering design of the plant: base case conditions (96%), reaction chemistry (96%), process flow diagram (85%), material balance (85%), energy balance (60%), property data (60%), equipment design (40%), major equipment list (30%) and labor requirements (10%).

3. Engineering design of the second distillation column (D-02, TCS column) in the process was completed. Specifications and results for process engineering design of the distillation column are reported including number of trays (equilibrium stages) required for the separation, respective feed tray location for each of the three feeds and stream data (flows and concentrations).

III. PLANS

Plans for the next reporting period are summarized below:

1. Continue chemical engineering analysis of the HSC process (Hemlock Semiconductor Corporation) for silicon.

2. For the preliminary process design, major efforts will be devoted to completion of base case conditions, reaction chemistry, process flow diagram and material balance. Additional activities will center on energy balance, equipment design, major equipment list and labor requirements.

3. Economic analysis of the HSC process will be initiated.

APPENDIX A1

PROCESS ENGINEERING: DESIGN SPECIFICATIONS FOR DISTILLATION, D-02

Date 1/18/82

Issue No. 1

1. Process Equipment Name Distillation, D-02 (TCS Column)
2. Process Equipment Function Separation of TCS (Trichlorosilane) and TET (Tetrachlorosilane).
3. Feed Specifications
   1. No. of Feeds 3
   2. No. of Feed Components 4
   3. Feed Components MCS, DCS, TCS, TET
   4. Feed Concentration See Item 7
   5. Feed Temperature See Item 7
   6. Feed Pressure See Item 7
   7. Light Key - LK Trichlorosilane (TCS)
   8. Heavy Key - HK Tetrachlorosilane (TET)
4. Distillate Specifications
   1. Recovery of Light Key (LK) in Distillate 97 %
   2. Concentration Spec. Low in TET
5. Bottoms Specifications
   1. Recovery of Heavy Key (HK) in Bottoms 97 %
   2. Concentration Spec. Low in MCS, DCS and TCS
6. General Specifications
   1. Pressure for Distillation 90 psia
   2. Condenser Type Total

APPENDIX A1

(Continued)

7. Feed Concentration

Component	Feed Concentration		
	Feed 1	Feed 2	Feed 3
1. SiH_3Cl , MCS	0.005	neglibile	neglibile
2. SiH_2Cl_2, DCS	0.100	0.167	0.0055
3. $SiHCl_3$, TCS	0.785	0.567	0.2552
4. $SiCl_4$, TET	0.110	0.266	0.7393
Total	1.000	1.000	1.0000
Temperature (F)	176	210	242
Pressure (Psia)	80	90	90
Mass Flow (lb-mole/hr)	230.23	15.25	174.33
Liquid Fraction	1.	1.	1.
Feed Source	Redistribution Reactor Effluent	CVD Reactor Recovery Unit Effluent	D-01 Bottoms

Note:

1. Feed concentration for feed 1 is from ref. A4, pg. 12
2. Feed concentration for feed 2 is from ref. A4, pg. 12
3. Feed concentration for feed 3 is from design of D-01

APPENDIX A2

PROCESS ENGINEERING: DESIGN RESULTS FOR DISTILLATION, D-02

Date 1/18/82

Issue No. 1

1. Process Equipment Name Distillation, D-02 (TCS Column)
2. Equipment Specifications
   1. No. of Equilibrium Trays N = 20
   2. No. of Equilibrium Feed Tray N_F = 9, 12, 15
   3. Tray Efficiency 63 %
   4. No. of Actual Trays N_{actual} = 32
   5. No. of Actual Feed Tray $N_{F,actual}$ = 16, 19, 24
   6. Tray Spacing 24 in.
   7. Type of Tray Single Pass Flow Seive Tray
   8. Column Diameter 5.5 ft.
   9. Column Height 75 ft.
   10. Reflux Ratio R = 2.0
   11. Design Temp. Top = 91 C
       Bottom = 126 C
   12. Design Pressure 90 psia
   13. Materials of Construction Steel
3. Product Specifications
   1. Feed Specifications
      1. Feed Concentration See Item 7 of Design Spec.
      2. Light Key - LK Trichlorosilane (TCS)
      3. Heavy Key - HK Tetrachlorosilane (TET)
   2. Distillate Specifications
      1. Recovery of Light Key (LK) in Distillate 97 %
      2. Concentration Spec. See Item 4
   3. Bottoms Specifications
      1. Recovery of Heavy Key (HK) in Bottoms 97 %
      2. Concentration Spec. See Item 4

APPENDIX A
(Continued)

4. Results for Stream Concentrations

Component		Concentration	
		Distillate	Bottom
1.	SiH_3Cl , MCS	0.004487	6.338×10^{-18}
2.	SiH_2Cl_2, DCS	0.103400	7.707×10^{-6}
3.	$SiHCl_3$, TCS	0.885637	0.04088
4.	$SiCl_4$, TET	0.006476	0.9591
	Total	1.000000	1.000000

Distillate : 256.53 lb-mole/hr

Bottom : 163.28 lb-mole/hr

Feed 1 : 230.23 lb-mole/hr

Feed 2 : 15.25 lb-mole/hr

Feed 3 : 174.33 lb-mole/hr

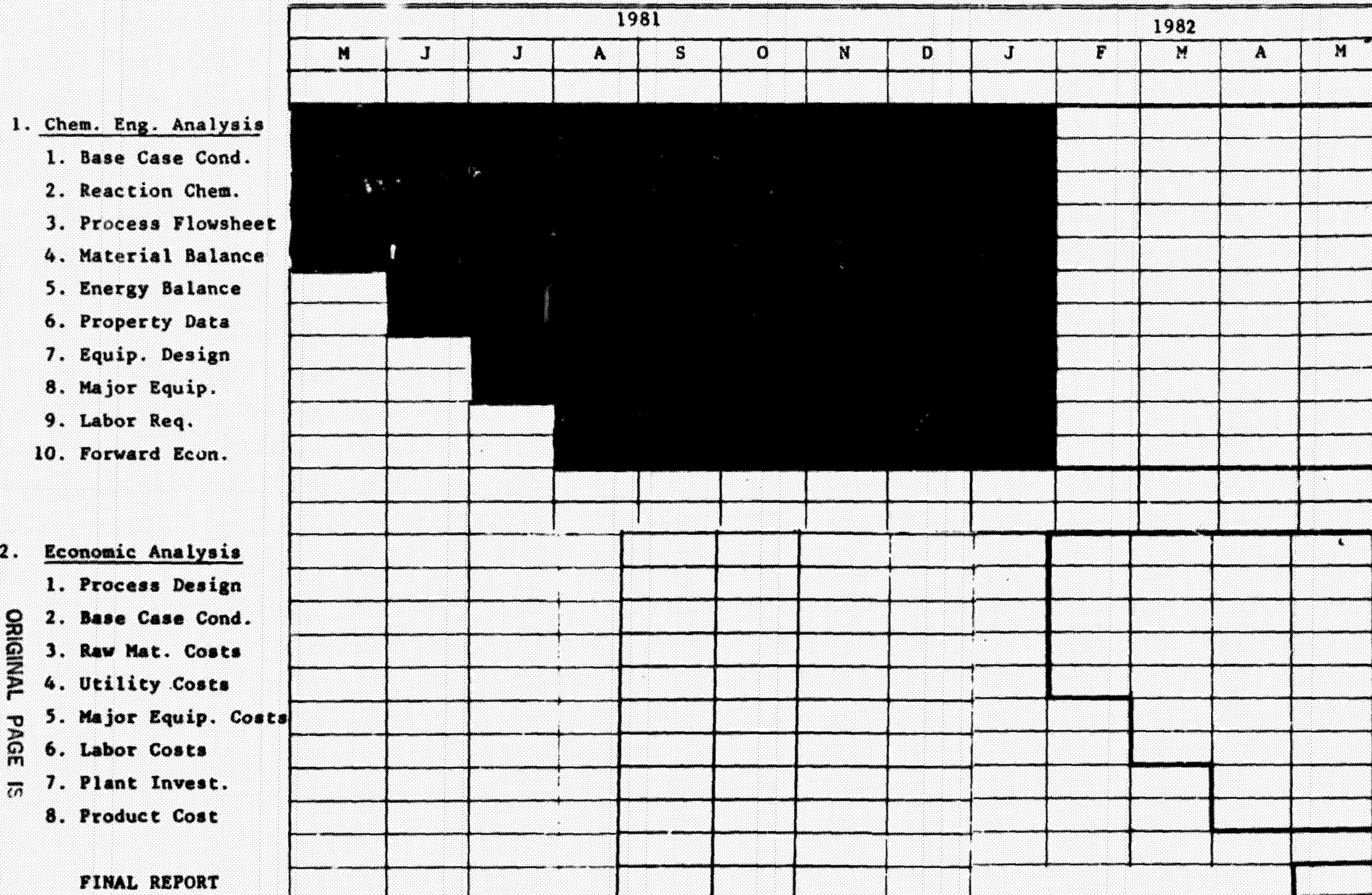
5. Results for Number of Trays

Reflux Ratio, R	No. of Equil. Trays, N	No. of Actual Trays N_{actual}
1	35 (16, 26, 30)	56
1.2	26 (13, 18, 21)	42
1.6	22 (10, 13, 16)	35
2	20 (9, 12, 15)	32
2.5	18 (7, 10, 13)	29
3.5	16 (6, 9, 12)	26
4.5	15 (6, 8, 11)	24

NOTE:

Numbers in parentheses give feed plate location. For case of R = 2, N_{F1}= 9, N_{F2}= 12 and N_{F3}= 15.

MILESTONE CHART



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